



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF
PREVENTION, PESTICIDES AND
TOXIC SUBSTANCES

MEMORANDUM

DATE: May 2, 2000

SUBJECT: Diclofop-methyl. List B Reregistration Case 2160.
PC Code 110902. Product and Residue Chemistry
Chapters for the Reregistration Eligibility
Decision (RED) Document. DP Barcode D265277.

FROM: Sheila Piper, Chemist
Chemistry and Exposure Branch I
Health Effects Division [7509C]

THRU: Francis B. Suhre, Branch Senior Scientist
Chemistry and Exposure Branch I
Health Effects Division [7509C]

TO: Christina Jarvis, Risk Assessor
Reregistration Branch 2
Health Effects Division [7509C]

The Product and Residue Chemistry chapters for the Diclofop-methyl RED are attached. The chapters were assembled by Dynamac Corporation under supervision of CEB1, HED. The data assessment has undergone secondary review in the Branch and has been revised to reflect Agency policies.

Several product chemistry data requirements remain outstanding. Provided the registrant submits the required data or submits complete updated product chemistry data packages, the Branch has no objections to the reregistration of diclofop-methyl with respect to product chemistry data requirements.

The qualitative nature of the residue(s) of diclofop-methyl in plants and animals have been satisfied. Additional data are needed to support existing enforcement methods used for tolerance enforcement (See details in OPPTS GLN 860.1300, this memo). The submitted field trial data for wheat and barley are adequate to reassess tolerances. Label revisions specify preharvest intervals (PHI's) of 66 days for barley

grain and 77 days for wheat grain. Storage stability data adequately supports the storage intervals of samples collected from the wheat and barley field trials and processing studies. Further details are provided in the endnotes to Table B in the Residue Chemistry chapter. The submitted confined rotational crop study is adequate pending submission of storage stability data.

The available ruminant feeding study suggests that tolerances should be established for diclofop and diclofop acid in milk and livestock (cattle, goats, horses, hogs, and sheep) commodities. The recommended tolerance levels are presented in "Tolerance Reassessment" section. There are no Codex MRLs for residues of diclofop-methyl, therefore, Codex/U.S. tolerance compatibility is not an issue.

With regard to dietary exposure assessment, the HED Metabolism Review Committee has determined the residue of concern for plants is diclofop-methyl and its metabolites, 2-[4-(2,4-dichlorophenoxy)phenoxy]propanoic acid and 2-[4-(2,4-dichloro-5-hydroxyphenoxy)phenoxy]propanoic acid and hydroxy conjugates. For animals, the residue of concern is diclofop-methyl and its metabolite, 2-[4-(2,4-dichlorophenoxy)phenoxy]propanoic acid. HED will conduct a dietary exposure assessment using the best available data, making conservative assumptions from metabolism data to estimate all residues of concern.

Attachment 1: Reregistration Eligibility Decision: Product Chemistry Considerations

Attachment 2: Reregistration Eligibility Decision: Residue Chemistry Considerations

cc: List B file, SF, RF, Dockter, C. Jarvis, R. Griffin, S. Piper, N. Seyed, R. Fricke, B. Shackelford; SRRD.

RDI: ChemSAC 4/26/00; F.B.Suhre 4/26/00;

S.Piper; CM2:Rm 810F:703-308-2717: Diclofop-methyl.

DICLOFOP-METHYL
PC Code 110902; Case No. 2160
DP Barcode D265277

Reregistration Eligibility Decision:

September 10, 1999

Contract No. 68-W-99-053

**Submitted to:
U.S. Environmental Protection Agency
Arlington, VA**

**Submitted by:
Dynamac Corporation
The Dynamac Building
2275 Research Boulevard
Rockville, MD 20850-3268**

DICLOFOP-METHYL

REREGISTRATION ELIGIBILITY DECISION:

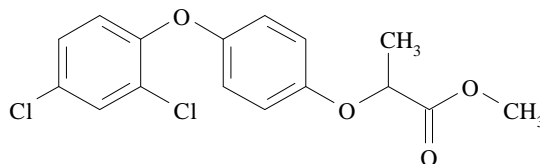
PRODUCT CHEMISTRY CONSIDERATIONS

PC Code 110902; Case No. 2160

DP Barcode D265277

DESCRIPTION OF CHEMICAL

Diclofop-methyl [methyl 2-(4-(2,4-dichlorophenoxy)phenoxy)propanoate] is an herbicide used on barley, wheat and golf courses.



Empirical Formula:	C ₁₆ H ₁₄ Cl ₂ O ₄
Molecular Weight:	341.19
CAS Registry No.:	51338-27-3
PC Code:	110902

IDENTIFICATION OF ACTIVE INGREDIENT

Diclofop-methyl is a colorless, crystalline solid with a melting point of 39-41 C, density of 1.30±0.05 g/cm³ at 40 C, octanol/water partition coefficient (P_{ow}) of 37800, and vapor pressure of 1.9 x 10⁻⁶ mm Hg at 20 C. Diclofop-methyl is practically insoluble in water (0.3 mg/100 mL), and is soluble in xylene (253 g/100 mL), acetone (249 g/100 mL), and ethanol (11 g/100 mL).

MANUFACTURING-USE PRODUCTS

A search of the Reference Files System (REFS) conducted on 11/26/99 identified a single diclofop-methyl manufacturing-use product (MP) registered under PC Code 110902: the AgrEvo USA Company 93% technical (T; EPA Reg. No. 45639-174). The AgrEvo 93% T was transferred 9/17/94 from Clariant Corporation (Hoechst Celanese Corporation; EPA Reg. No. 8340-21). AgrEvo is the registered U.S. agent of Hoechst Schering AgrEvo S.A. Because diclofop-methyl is a list B chemical, only the 93% T/TGAI is subject to a reregistration eligibility decision.

REGULATORY BACKGROUND

The Diclofop-methyl Phase 4 Review dated 2/12/91 by J. Smith determined that additional data were required concerning starting materials and manufacturing processes, discussion of formation of impurities, preliminary analysis, certified limits, enforcement analytical methods, stability, dissociation constant, and solubility (OPPTS 830.1550, .1600, .1620, .1670, .1700, .1750, .1800, .6313, .7370, and .7840); all other product chemistry data submissions were determined to be acceptable for Phase 5 review.

In addition, data were required concerning the potential for formation of nitrosamines in diclofop-methyl, and a Data Call-In (DCI) notice dated 4/2/90 required data concerning the potential for formation of polychlorinated dibenzo-*p*-dioxin or dibenzofuran contaminants. Nitrosamine and dioxin analyses demonstrate that N-nitroso compounds and polychlorinated dibenzo-*p*-dioxins and dibenzofurans have not been detected in diclofop-methyl at the respective Agency-specified LOQs.

The current status of the product chemistry data requirements for the diclofop-methyl T/TGAI is presented in the attached data summary table.

CONCLUSIONS

Pertinent product chemistry data requirements remain unfulfilled for the AgrEvo 93% T/TGAI. Additional data are required concerning product identity & composition, discussion of formation of impurities, certified limits, enforcement analytical method, pH, UV/visible absorption, and vapor pressure (OPPTS 830.1550, .1670, .1750, .1800, .7000, .7050, and .7950). Provided that the registrant submits the data required in the attached data summary table for the diclofop-methyl T/TGAI, and either certifies that the suppliers of beginning materials and the manufacturing process have not changed since the last comprehensive product chemistry review or submits a complete updated product chemistry data package, the Agency has no objections to the reregistration of diclofop-methyl with respect to product chemistry data requirements.

Case No. 2160
Chemical No. 110902

Case Name: Diclofop-methyl
Registrant: AgrEvo USA Company
Product(s): 93% T (EPA Reg. No. 45639-174)

PRODUCT CHEMISTRY DATA SUMMARY

Guideline Number	Requirement	Are Data Requirements Fulfilled? ¹	MRID Number ²
830.1550	Product identity and composition	N ³	00068748, CSF 3/29/93
830.1600	Description of materials used to produce the product	Y	40623104
830.1620	Description of production process	Y	40623104
830.1670	Discussion of formation of impurities	N ⁴	40623105
830.1700	Preliminary analysis	Y	42218801 ⁵ , 42156901 ⁶ , 42717001 ⁷ , 43492201 ⁸
830.1750	Certified limits	N ³	00068748, CSF 3/29/93
830.1800	Enforcement analytical method	N ⁹	
830.6302	Color	Y	41573301
830.6303	Physical state	Y	41573301
830.6304	Odor	Y	41573301
830.6313	Stability to normal and elevated temperatures, metals, and metal ions	Y	42796401 ¹⁰ , 43396701 ⁸
830.7000	pH	N ¹¹	
830.7050	UV/Visible absorption	N ¹²	
830.7200	Melting point/melting range	Y	41573301
830.7220	Boiling point/boiling range	N/A ¹³	
830.7300	Density/relative density/bulk density	Y	41573301
830.7370	Dissociation constants in water	Y	42461501 ¹⁴
830.7550	Partition coefficient (n-octanol/water), shake flask method	Y	40806305
830.7840	Water solubility: column elution method; shake flask method	Y	42796401 ¹⁰
830.7950	Vapor pressure	N ¹⁵	40806304

¹ Y = Yes; N = No; N/A = Not Applicable.

² **Bolded** references were determined to be acceptable for Phase 5 review in the Diclofop-methyl Phase 4 Review dated 2/12/91 by J. Smith, and were reviewed under D252789, 11/26/99, K. Dockter; and all other references were reviewed as noted.

³ A revised CSF must be submitted reflecting the current registrant and EPA registration number.

⁴ Additional discussion is required concerning the potential for formation of post-production impurities resulting from degradation of the product, cross-contamination from manufacturing equipment, or migration of packaging

materials.

⁵ CBRS Nos. 9672 and 10001, D176227 and D179259, 9/16/93, P. Deschamp.

⁶ CBRS No. 9252, D173293, 6/2/92, S. Funk.

⁷ CBRS No. 11752, D190377, 6/15/93, S. Funk.

⁸ CBRS Nos. 14579 and 14908, D208353 and D210557, 5/8/95, K. Dockter.

⁹ The registrant committed to generate a new study (Phase 4 Review).

¹⁰ CBRS No. 11989, D192146, 8/5/93, K. Dockter.

¹¹ An aqueous suspension of the chemical substance should be tested [see 830.7000 (c)(2)].

¹² The OPPTS Series 830, Product Properties Test Guidelines require data pertaining to UV/visible absorption for the PAI.

¹³ Data are not required because the TGAI is a solid at room temperature.

¹⁴ CBRS No. 10613, D182830, 10/23/92, A. Aikens.

¹⁵ The test method and test substance purity must be specified.

AGENCY MEMORANDA CITED IN THIS DOCUMENT

CBRS No(s): 9252
DP Barcode(s): D173293
Subject: Determination of Polychlorinated Dibenzo-p-dioxins and Dibenzofurans in Diclofop-Methyl (List B, Case 2160, Chemical 110902).
From: S. Funk
To: B. Shackleford/T. Luminello
Dated: 6/2/92
MRID(s): 42156901

CBRS No(s): 10613
DP Barcode(s): D182830
Subject: Diclofop-methyl Reregistration. Product Chemistry Guideline 63-10 (Dissociation Constant). Response to Phase 4 Review.
From: A. Aikens
To: L. Deluise/T. Luminello
Dated: 10/23/92
MRID(s): 42461501

CBRS No(s): 11752
DP Barcode(s): D190377
Subject: Determination of Polychlorinated Dibenzo-p-dioxins and Dibenzofurans in Diclofop-Methyl (List B, Case 2160, Chemical 110902).
From: S. Funk
To: K. Davis/T. Luminello
Dated: 6/15/93
MRID(s): 42717001

CBRS No(s): 11989
DP Barcode(s): D192146
Subject: Diclofop-methyl Reregistration. Hoechst Celanese's 6/1/93 Response [63-8 & -13 data for 8340-21] to Our 2/12/91 Phase 4 Review.
From: K. Dockter
To: T. Luminello
Dated: 8/5/93
MRID(s): 42796401

CBRS No(s): 9672 and 10001
DP Barcode(s): D176227 and D179259
Subject: Reregistration of Diclofop-methyl. Product Chemistry Considerations. List B Case No. 2160. Chemical No. 110902.
From: P. Deschamp
To: T. Luminello
Dated: 9/16/93
MRID(s): 42218801

CBRS No(s): 14579 and 14908
DP Barcode(s): D208353 and D210557
Subject: Diclofop-methyl. AgrEvo 10/4/94 & 12/21/94 Responses [63-13 & 62-1 (Nitrosamine) data] to Agency 4/1/94 Letter; Re: K. Dockter 8/5/93 & P. Deschamp 9/16/93 Memoranda.
From: K. Dockter
To: K. Davis/T. Luminello
Dated: 5/8/95
MRID(s): 43396701 and 43492201

DP Barcode(s): D252789
Subject: Product Chemistry data in support of the Reregistration of Diclofop-methyl.
From: K. Dockter
To: Christina Jarvis
Dated: 11/26/99
MRID(s): 00068748, 40623104, 40623105, 40806304, 40806305, and 41573301

PRODUCT CHEMISTRY CITATIONS

Bibliographic citations include only MRIDs containing data which fulfill data requirements.

References (cited):

00068748 American Hoechst Corporation (1977) Basic Manufacturing Process, Chemical Composition and Physical Data of Hoelon(TM). Unpublished study received Sep 15, 1977 under 8340-EX-2; CDL: 096359-A.

40623104 Friedrich (1987) HOE 023408: Diclofop-methyl Technical: Description of Beginning Materials and Manufacturing Process: Laboratory Project No. PROD 87/1: A 36811. Unpublished compilation prepared by Hoechst Aktiengesellschaft. 21 p.

40623105 Sarafin, R. (1987) HOE 023408: Discussion of the Formation of Impurities in the Technical Grade Substance: Laboratory Project No. (B)120/87. Unpublished compilation prepared by Hoechst Aktiengesellschaft. 21 p.

40806304 Grewer (1988) Determination of Vapor Pressure as a Function of Temperature of Diclofop-methyl (HOE 023408): Translation of Document No. A37786: Project ID. A37875. Unpublished study prepared by Hoechst AG. 6 p.

40806305 Dorn, E. (1979) Diclofop-methyl (HOE 23408) - Partition Coefficient (P) in the System N-Octanol/Water: Project ID. A18266. Unpublished study prepared by Hoechst AG. 14 p.

41573301 Maier; Rexer, K. (1990) Physical and Chemical Characteristics of Diclofop-methyl Technical (HOE 023408 00 ZC95 0001): Lab Project Number: A 42773: SUM/0001: WIR/0035. Unpublished study compared by Hoechst Aktiengesellschaft. 13 p.

42156901 Ehamann, J. (1961) Sampling and Analytical Program Regarding the Content of Polychlorinated Dibenzo-p-Dioxin (Dioxin) and Dibenzofuran (Furan) Contaminants of Diclofop-methyl: Hoelon Technical: Lab Project Number: A 46823: SP 91/HOE 01. Unpublished study prepared by Institut Biocontrol fur Chemische und Biologische. 300 p.

42218801 Gorlitz, G.; Gubert, M. (1992) Diclofop-methyl (HOE 23408): Analysis of Seven Typical Production Batches: Lab Project Number: A 47112: CP91/100. Unpublished study prepared by Hoechst Ag. 92 p.

42461501 Weidenauer, M.; Mollard, L. (1992) Determination of the Dissociation Constant of Diclofop (Hoe 021079) According to the OECD Guideline for Testing of Chemicals Nr. 112, Adopted on 12 May, 1981: Lab Project Number: BE-P-1-92-PK-01-BG. Unpublished study prepared by Battelle Europe. 22 p.

42717001 Ehamann, J. (1993) Sampling and Analytical Program Regarding the Content of Polychlorinated Dibenzo-P-Dioxin (Dioxin) and Dibenzofuran (Furan) Contaminants of Diclofop-methyl Hoelon Technical (HOE 023 408): Amendment to the Final Report: Lab Project Number: SP 91/HOE 01. Unpublished study prepared by Institut fur Chemische und Biologische Untersuchungen. 72 p.

42796401 Pesselman, R. (1993) Series 63 Product Chemistry Determinations of Diclofop-methyl (Solubility and Stability): Lab Project Number: HWI 6187-135. Unpublished study prepared by Hazleton Wisconsin, Inc. 71 p.

43396701 Netzband, D. (1994) The Determination of the Stability of Diclofop-methyl Technical: Lab Project Number: 1140/94/01/1. Unpublished study prepared by AgrEvo Research Center.

27 p.

43492201 Repenthin, W. (1994) Determination of the Total Content of N-Nitroso compounds in Seven Technical Batches of Diclofop-methyl (HOE 023 408): Lab Project Number: AZ 152 094: A 53405: SPA 06/94. Unpublished study prepared by Schering AG. 20 p.

DICLOFOP-METHYL

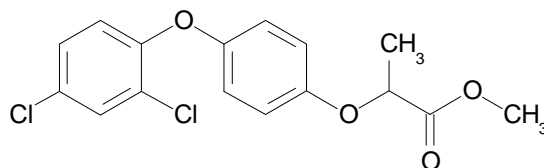
REREGISTRATION ELIGIBILITY DECISION

RESIDUE CHEMISTRY CONSIDERATIONS

PC Code 110902; Case 2160

TABLE OF CONTENTS	page
INTRODUCTION	1
REGULATORY BACKGROUND	1
SUMMARY OF SCIENCE FINDINGS	2
OPPTS GLN 860.1200: Directions for Use	2
OPPTS GLN 860.1300: Nature of the Residue in Plants	4
OPPTS GLN 860.1300: Nature of the Residue in Livestock	4
OPPTS GLN 860.1340: Residue Analytical Methods	5
OPPTS GLN 860.1360: Multiresidue Method Testing	6
OPPTS GLN 860.1380: Storage Stability Data	7
OPPTS GLN 860.1500: Magnitude of the Residue in Crop Plants	7
OPPTS GLN 860.1520: Magnitude of the Residue in Processed Food/Feed	8
OPPTS GLN 860.1480: Magnitude of the Residue in Meat, Milk, Poultry, and Eggs	8
OPPTS GLN 860.1400: Magnitude of the Residue in Water, Fish, Irrigated Crops	10
OPPTS GLN 860.1460: Magnitude of the Residue in Food-handling Establishments ...	10
OPPTS GLNs 860.1850/860.1900: Confined/Field Accumulation in Rotational Crops	10
TOLERANCE REASSESSMENT SUMMARY	18
Tolerances To Be Listed Under 40 CFR §180.385(a)(1):	18
Tolerances Needed Under 40 CFR §180.385(a)(1)	19
Tolerances Needed Under 40 CFR §180.385(a)(2)	19
CODEX HARMONIZATION	20
AGENCY MEMORANDA	21
RESIDUE CHEMISTRY CITATIONS	24

DICLOFOP-METHYL



REREGISTRATION ELIGIBILITY DOCUMENT

RESIDUE CHEMISTRY CONSIDERATIONS

PC Code 110902; Case 2160

INTRODUCTION

Diclofop-methyl [methyl-2-(4-(2,4-dichlorophenoxy)phenoxy)propanoate] is a selective postemergence herbicide registered for the control of annual grasses. The reregistration of diclofop-methyl on only two food/feed crops, barley and wheat, is being supported by AgrEvo USA, a company of Hoechst and Schering. Diclofop-methyl is sold in the United States as Hoelon® 3EC Herbicide and may be applied preplant-incorporated or preemergence for winter wheat, and postemergence for wheat and barley using ground or aerial equipment.

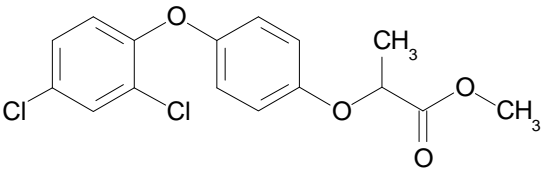
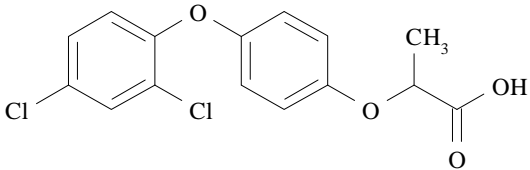
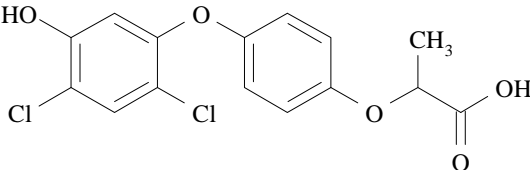
REGULATORY BACKGROUND

Diclofop-methyl is a List B reregistration chemical and was the subject of a Phase 4 Review (J. Smith, 2/12/91). The Phase 4 Review summarized the available data for each residue chemistry guideline and specified the additional data required for reregistration purposes. Data Call-In (DCI) Notices for diclofop-methyl were issued by the Agency on 6/5/91 and 10/13/95. The basic producer has submitted several residue chemistry studies since issuance of the Phase 4 Review, and most of these studies have been evaluated by HED. The information contained in this document outlines the current Residue Chemistry Science Assessments with respect to the reregistration of diclofop-methyl.

Tolerances have been established for the combined residues of diclofop-methyl and its metabolites 2-[4-(2,4-dichlorophenoxy)phenoxy]propanoic acid and 2-[4-(2,4-dichloro-5-hydroxyphenoxy)phenoxy]propanoic acid at 0.1 ppm in/on barley grain and straw, lentils, dry peas, and wheat grain and straw [40 CFR §180.385(a)]. The chemical names and structures of diclofop-methyl and its presently regulated metabolites are depicted in Figure A.

Figure A. Chemical Names and Structures of Diclofop-Methyl and its Metabolites Which are Presently

Included in the Tolerance Expression as Listed in 40 CFR §180.385(a).

Common Name (Codes) Chemical Name	Chemical Structure
Diclofop-methyl (HOE-023408; AE F023408) methyl 2-[4-(2,4-dichlorophenoxy)phenoxy] propanoate	
Diclofop acid (HOE-021079; AE F021079) 2-[4-(2,4-dichlorophenoxy)phenoxy] propanoic acid	
Hydroxy diclofop (HOE-038182; AE F038182) 2-[4-(2,4-dichloro-5-hydroxyphenoxy) phenoxy] propanoic acid	

SUMMARY OF SCIENCE FINDINGS

OPPTS GLN 860.1200: Directions for Use

A search of the Agency's Reference Files System (REFS) on 1/11/00 indicates that there are two active diclofop-methyl end-use products (EPs) with feed/food uses which are registered to AgrEvo USA Company; these EPs are listed in Table A1. It is noted that one of these products, EPA Reg. No. 45639-177, has been canceled.

Table A1. Diclofop-Methyl End-Use Products With Food/Feed Uses Registered to AgrEvo USA.

EPA Reg No.	Label Acceptance Date As Per 1/11/00 REFS Search	Formulation Class	Product Name
45639-173 ¹	12/99 ²	3 lb/gal EC	Hoelon® 3EC Herbicide
45639-177 ³	2/96	3 lb/gal EC	Hoelon® 3EW Herbicide

¹ Includes the following SLNs with non-food/feed uses: AL900011, AR930002, FL960001, GA950009, LA980008, MS910019, NC910001, OK930003, SC900003, SC990007, TN930008, and TX920009.

² The actual available label for EPA Reg. No. 45639-173 was dated 2/99.

³ Canceled as per USEPA/OPP Chemical Database (1/13/00).

The Hoelon 3EC herbicide may be applied preplant-incorporated and preemergence (wheat only), or postemergence for wheat and barley depending on the crop and the grass species being controlled. The 3 lb/gal EC formulation is registered for a single broadcast post-emergence spray in barley at 0.5-1 lb ai/A. Post-emergence application may be made beginning at the 1-3 leaf stage until the 2-tiller stage of barley growth. The 3 lb/gal EC formulation for wheat is registered for (i) pre-plant incorporated application on winter wheat at 0.75-1 lb ai/A; (ii) pre-emergence application on winter wheat at 0.75-1 lb ai/A; and (iii) post-emergence application at the 1-3 leaf stage until the 2-tiller stage of wheat growth. Application for barley or wheat may be made using ground (≥ 10 gal water/A) or aerial (≥ 5 gal water/A) equipment.

- Label revisions specify preharvest intervals (PHI's) of 66 days for barley grain and 77 days for wheat grain.
- The label states that livestock may not graze on treated fields, and includes a restriction against the harvest of forage, hay or straw from treated fields prior to grain harvest. CBRs notes that these label restrictions are no longer considered practical, and that residue data must be generated demonstrating diclofop-methyl in barley hay and straw.
- Based on the confined rotational crop studies, a 120-day restriction is required for planting root and tuber vegetables, leafy vegetables and small grains as rotational crops in diclofop-methyl treated soils.

A comprehensive summary of the registered use patterns of diclofop-methyl, based on the product label registered to AgrEvo USA, is presented in Table A2. A tabular summary of the residue chemistry science assessments for reregistration of diclofop-methyl is presented in Table B. The conclusions listed in Table B regarding the reregistration eligibility of diclofop-methyl food/feed uses are based on the use patterns registered by the basic producer, AgrEvo USA Company. When end-use product DCIs are developed (e.g., at issuance of the RED), RD should require that all end-use product labels (e.g., MAI labels, SLNs, and products subject to the generic data exemption) be amended such that they are consistent with the basic producer's labels.

OPPTS GLN 860.1300: Nature of the Residue in Plants

The qualitative nature of the residue in plants is acceptable based on the wheat metabolism study. Wheat plants at the early tillering stage were treated, in separate experiments, with [¹⁴C]diclofop-methyl radiolabeled in the dichlorophenyl and dioxyphenyl rings at a 1x label rate. Immature crop parts were collected from day 0 to day 78, and mature crop parts were collected at normal harvest (101 days after treatment). The parent, diclofop-methyl, and the diclofop acid metabolite were not detected in either mature straw or mature grain. There was no evidence for extensive cleavage of the phenyl ether linkage. Based primarily on residue characterization and identification of immature wheat shoots, HED concluded that diclofop-methyl is converted to diclofop acid, and diclofop is hydroxylated on the dichlorophenyl ring. The latter may form conjugates (straw), and/or undergo degradation and re-incorporation into bound molecules (grain) that are released by enzyme treatment and vigorous acid hydrolysis. The HED Metabolism Assessment Review Committee (Memo, 4/7/00, S.Piper) questioned whether the enforcement method would detect metabolites characterized as hydroxy metabolite conjugates (M5 and M7) and indicated that the registrant must demonstrate whether the enforcement method will determine M5 and M7. The MARC concluded that the residues of concern for wheat and barley are diclofop-methyl, diclofop acid [2-[4-(2,4-dichlorophenoxy) phenoxy]propanoic acid], and hydroxy diclofop [2-[4-(2,4-dichloro-5-hydroxyphenoxy) phenoxy]propanoic acid] and its conjugates. The MARC may reassess the conclusion regarding the residues of concern in plants upon submission of additional information.

OPPTS GLN 860.1300: Nature of the Residue in Livestock

The qualitative nature of the residue in animals is acceptable based on the ruminant and poultry metabolism studies. Following HED reviews of these studies, it was concluded that diclofop-methyl is metabolized similarly in ruminant and poultry. The residues of concern for both ruminants and poultry are diclofop-methyl and diclofop acid, free and conjugated. Regulation of hydroxy diclofop in animal matrices is not necessary since its concentration in animal tissues is relatively low. The results of these metabolism studies triggered the need for poultry and ruminant feeding studies (as well as appropriate residue analytical methods) to determine the need for tolerances on milk, eggs, and animal tissues.

In the ruminant study (MRID 42450101), separate lactating goats were orally dosed once a day for three consecutive days with either [dichlorophenoxy-¹⁴C]diclofop-methyl or [dioxyphenyl-¹⁴C]diclofop-methyl at feeding levels of 11.5 ppm and 11.2 ppm, respectively, in the diet. The feeding levels represent 0.4x and 0.8x the maximum theoretical dietary burdens of 13.8 ppm and 29.5 ppm for beef and dairy cattle, respectively; see OPPTS GLN 860.1480 section for calculation of dietary burdens for diclofop-methyl. At sacrifice, total radioactive residues (TRR) were determined, and the maximum TRRs were 1.4 ppm in milk, 0.28 ppm in fat, 0.27 ppm in muscle, 2.4 ppm in liver, 15.2 ppm in kidney, and 3.7 ppm in blood. The majority of the radioactive residues in the examined tissues was adequately characterized and identified. The

parent, diclofop-methyl, was not detected in any goat matrix. The principal residues identified were free diclofop acid and various lipid conjugates of diclofop acid. Minor identified metabolites included a hydroxy- and a phenolic- derivative of the parent compound.

In the poultry study (MRID 43529601), separate groups of laying hens were orally administered for five consecutive days with either [dichlorophenoxy-¹⁴C]diclofop-methyl or [dioxiphenyl-¹⁴C]diclofop-methyl at nominal rates of 10 ppm, based on feed consumption. The feeding level represents >100x the maximum theoretical dietary burden of 0.09 ppm for poultry. Appropriate tissues were collected and analyzed for determination of TRR. The TRR levels were 0.3-0.5 ppm in egg yolks, 0.002 ppm in egg whites, 0.10-0.16 ppm in whole eggs, 0.19-0.31 ppm in fat, 0.7-0.9 ppm in kidney, 0.6-0.8 ppm in liver, and 0.01-0.02 ppm in muscle. The TRR levels were similar in comparable tissues from both radiolabel treatments. The parent, diclofop-methyl, was not detected in any poultry matrix. The principal residue identified was the diclofop acid, free and conjugated forms, which comprised 98% TRR in egg yolk, 4% TRR in muscle, 44% TRR in fat, and 70% TRR in liver. The only other residue component identified was diclofop phenol which accounted for 1.5% TRR in the liver of hen treated with [dichlorophenoxy-¹⁴C]diclofop-methyl.

The HED Metabolism Assessment Review Committee (Memo, 4/7/00, S.Piper) determined the residue of concern for animals is diclofop-methyl and its metabolite diclofop acid (free and conjugated).

OPPTS GLN 860.1340: Residue Analytical Methods

Plant methods: The Pesticide Analytical Manual (PAM) Volume II lists a GLC/ECD method, designated as Method I, for the enforcement of plant commodity tolerances. Method I determines residues of the parent and hydroxy diclofop. It does not specifically state that it determines diclofop acid but this acid metabolite would likely be methylated to the parent in the methylation step. Method I additionally determines residues of a phenol metabolite, 4-(2, 4-dichlorophenoxy)phenol, which is converted to 4-(2, 3-dichlorophenoxy)anisole in the methylation step. The phenol metabolite is a soil metabolite and is not a residue of concern in plants. The stated detection limit of Method I is 0.05 ppm.

The data-collection method used to generate barley and wheat residue data was a GLC/ECD method, designated as Method HRAV-14. Briefly, residues of parent and its metabolites are extracted from the sample by refluxing/stirring with 20% aqueous triethanolamine. This process converts diclofop-methyl to diclofop-free acid. At the end of the extraction/hydrolysis, the mixture is allowed to cool before adding acetone to precipitate unwanted organic matter. The hydrolysate is then centrifuged, the supernatant is decanted, and residues are extracted twice by stirring with acetone:water (1:1, v:v), followed by another centrifugation step. The combined extracts are partitioned with dichloromethane:hexane (3:1, v:v). The latter organic extract is taken to dryness, and residues are derivatized using diazomethane. Clean up can be achieved using

a Florisil SPE, eluting with 5% ethyl acetate in hexane. The eluates are evaporated to near dryness under vacuum, then re-solubilized in hexane prior to GLC/ECD analysis. The reported detection limit is 0.05 ppm for diclofop acid and hydroxy-diclofop. The concurrent method recovery data indicate that Method HRAV-14 is adequate for data collection.

Animal methods: The registrant proposes a GC/ECD (or MSD) method, designated as Method BL/01/95 version 2) for the enforcement of the required animal commodity tolerances. The method determines residues of diclofop-methyl and diclofop acid (free and lipid conjugates) in meat, milk, and eggs. The method includes procedures for hydrolysis of diclofop acid conjugates and derivatization of residues to diclofop-methyl. The validated LOQ for residues of diclofop-methyl and diclofop-acid in liver, fat, and eggs is 0.05 ppm. The LOQ for residues of diclofop-methyl and diclofop-acid in milk is 0.01 ppm. The method was radiovalidated using samples from the ruminant metabolism study and was adequately subjected to an independent laboratory validation (ILV). The Branch will forward Method BL/01/95 version 2 to ACB/BEAD for a method validation.

A brief description of Method BL/01/95 version 2 follows. Residues of diclofop-methyl and diclofop acid in kidney, liver, and muscle are extracted with ethyl acetate (EtOAc), centrifuged, and concentrated to dryness. Residues are re-constituted in methyl tert-butyl ether, derivatized in the presence of methyl iodide and tetra-butyl ammonium hydroxide to form diclofop-methyl, cleaned-up on a silica SPE column, and analyzed by GC/ECD. In fat, residues of diclofop-methyl, diclofop-acid, and their lipid conjugates are hydrolyzed with 1 N HCl to convert the entire residue to diclofop acid. Residues are extracted into hexane:acetone (95:5, v:v), centrifuged, concentrated, methylated, and determined in the same manner as described above, except that analysis is conducted using GC/MSD (quantitation ion, m/z 340).

Residues of diclofop-methyl, diclofop-acid, and their lipid conjugates in milk and eggs are acid hydrolyzed with concentrated or 1 N HCl, and sequentially extracted into acetone, EtOAc, and hexane, centrifuged, and the extracts combined. The combined extracts are shaken (aqueous layer discarded), residues in the organic layer are dried over sodium sulfate, and concentrated to dryness. Residues are then derivatized and determined following the procedures described above using GC/ECD.

OPPTS GLN 860.1360: Multiresidue Method Testing

The reregistration requirements for multiresidue methods data are fulfilled. The 2/97 FDA PESTDATA database (PAM Volume I, Appendix I) indicates that diclofop-methyl is completely recovered (>80%) using Multiresidue Methods Sections 302 (Luke Method; Protocol D), 303 (Mills, Onley, and Gaither; Protocol E, nonfatty) and 304 (Mills; Protocol E, fatty food).

The metabolites diclofop acid and hydroxy diclofop were also subjected to applicable multiresidue methods tests. Recoveries using Protocol B were variable for diclofop acid and

hydroxy diclofop. Small (<50%) to complete (>80%) recoveries were obtained from soybeans and wheat grain for diclofop acid; no recoveries (0%) to small (<50%) recoveries were obtained from soybeans and wheat grain for hydroxy diclofop.

OPPTS GLN 860.1380: Storage Stability Data

Data (MRIDs 42442801-05; 42857501) are available indicating that residues of diclofop-methyl, diclofop acid, and hydroxy diclofop are stable under frozen (<0 C) conditions in/on wheat matrices (grain, straw, bran, shorts, and flour) for at least 25 months. These data adequately support the storage conditions and intervals of samples collected from the wheat and barley field trial and processing studies where samples were stored frozen for a maximum of 25 months (wheat processed fractions).

Data (MRID 44915001) are also available indicating that residues of diclofop-methyl and diclofop acid are stable in animal commodities (beef muscle, milk, poultry liver, and eggs) stored at <-10 C for at least 23 months. These data adequately support the storage conditions and intervals of samples collected from the animal feeding studies in which samples were stored frozen from a maximum of 16 months.

OPPTS GLN 860.1500: Magnitude of the Residue in Crop Plants

Adequate field residue studies, depicting diclofop-methyl residues of concern, in/on the commodities of barley and wheat have been submitted and evaluated. An adequate number of field trials were conducted in the respective crop growing areas using a representative EC formulation of diclofop-methyl at the maximum labeled use rate.

A barley field study (MRID 42442802) indicates that the combined residues of diclofop-methyl, diclofop acid and hydroxy diclofop were nondetectable (<0.10 ppm) in/on barley grain and straw harvested 66-98 days following a single postemergence foliar broadcast application of the 3 lb/gal EC formulation at 2x the maximum registered rate. In another barley field study (MRID 44896102), the maximum combined residues were 5.653 ppm in/on barley hay harvested 33-50 days following a single postemergence foliar broadcast application of the EC formulation at 1x.

A wheat field study (MRID 42442801) indicates that the combined residues of diclofop-methyl, diclofop acid, and hydroxy diclofop were nondetectable (<0.10 ppm) in/on wheat grain and straw harvested 210-257 days following a single preplant incorporated or pre-emergence application, or 77-119 days following a single postemergence application of the 3 lb/gal EC formulation at 1x. In another wheat field study (MRID 44896101), the maximum combined residues were 11.878 ppm in/on wheat forage harvested 10-26 days and 0.913 ppm in/on wheat hay harvested 45-66 days following a single postemergence foliar broadcast application of the EC formulation at 1x. Adequate aspirated wheat grain fractions (grain dust) are available, and based on the use pattern

and submitted residue data, a tolerance is not necessary for this RAC.

OPPTS GLN 860.1520: Magnitude of the Residue in Processed Food/Feed

Acceptable barley and wheat processing studies (MRIDs 424428-01-05; 42857501) have been submitted and evaluated. These processing studies indicate that residues of diclofop-methyl and its regulated metabolites do not concentrate in the respective processed fractions of barley and wheat processed from grains which were treated at an exaggerated rate (~5x).

OPPTS GLN 860.1480: Magnitude of the Residue in Meat, Milk, Poultry, and Eggs

An acceptable ruminant feeding study (MRID 44178001-02) has been submitted and evaluated. Four groups of lactating dairy cows were dosed orally *via* capsules for 28 consecutive days with diclofop-methyl at levels equivalent to 0.11, 0.33, 1.1, and 25.0 ppm in the diet. These dose levels approximate 0.004x, 0.01x, 0.04x, and 0.85x the theoretical dietary burden of 29.5 ppm for dairy cattle; the calculation of the maximum theoretical dietary burdens of cattle, poultry, and swine for diclofop-methyl is presented in the table below. Samples of collected milk and cow tissues were analyzed using Method BL/01/95 version 2 which determines combined residues of diclofop-methyl and diclofop acid following hydrolysis of conjugate residues of diclofop acid and conversion of released residues to the parent. Quantifiable residues were detected in milk from all dose groups. Residues in whole milk plateaued by Day 4 at all dose levels. The maximum combined residues in whole milk were 0.023, 0.114, 0.212, and 2.759 ppm for the 0.004x, 0.01x, 0.04x, and 0.85x dose groups, respectively. Diclofop-methyl residues concentrated in milk fat, with residues in cream being 2.4-3.4x higher than in whole milk. For the 0.85x-dose group, residues were 5.11-5.79 ppm in cream (Day 21). Residues in skim milk were <LOQ except for one low-dose cow (0.027 ppm) and for the three cows in the highest dose group (0.024-0.043 ppm). In tissues, residues were highest in kidney and lowest in muscle. For the 0.85x dose group, the combined residues were 12.2-23.3 ppm in kidney, 3.89-6.12 ppm in liver, 0.748-0.851 ppm in fat, and 0.316-0.568 ppm in muscle. The results of the ruminant feeding study suggest that tolerances are required for milk and ruminant tissues. The recommended tolerance levels are presented in the “Tolerance Reassessment” section.

Calculation of the maximum theoretical dietary burdens of cattle, poultry, and swine for diclofop-methyl.

Feed Commodity	% Dry Matter	% Diet ¹	Tolerance (ppm) ²	Dietary Contribution (ppm) ³
Beef Cattle				
Wheat forage	25	25	12.0	12.0

Barley hay	88	25	6.0	1.70
Wheat grain	89	50	0.1	0.06
Total Burden				13.8
Dairy Cattle				
Wheat forage	25	60	12.0	28.8
Barley hay	88	10	6.0	0.68
Wheat grain	89	30	0.1	0.03
Total Burden				29.5
Poultry and Swine				
Wheat grain	89	80	0.1	0.09
Total Burden				0.09

¹ Table 1 (August 1996).

² Current or reassessed tolerance from the Diclofop-Methyl Reregistration Eligibility Decision (RED).

³ Contribution = [tolerance / % DM (if cattle)] X % diet).

A feeding study on swine is not available. HED will translate the residue data from the ruminant feeding study using residues obtained from the 1.1 ppm dosing level for determination of tolerances required for hog commodities.

An acceptable poultry feeding study (MRID 44178001-02) has been submitted and evaluated. The results of the study suggest that tolerances are not required for diclofop-methyl residues of concern in eggs and poultry tissues. There is no reasonable expectation of finding quantifiable diclofop-methyl residues of concern in eggs, fat, meat, and meat byproducts of poultry [40 CFR 180.6(a)(3)]. In the poultry study, three groups of laying hens were dosed with diclofop-methyl by capsule for 28 consecutive days at levels equivalent to 0.1, 0.3, and 1.0 ppm in the diet. These dose levels approximate 1x, 3x, and 11x the maximum theoretical dietary burden of 0.09 ppm for poultry. Samples of collected eggs and poultry tissues were analyzed using Method BL/01/95 version 2. For the high-dose group (11x), the maximum combined residues in eggs were attained by Day 1 at ≤ 0.155 ppm. Residues in eggs declined to ≤ 0.073 ppm by Day 4 and were $< \text{LOQ}$ (< 0.05 ppm) at all subsequent sampling intervals, indicating that residues do not accumulate in eggs. Residues in eggs from the 1x and 3x groups on Days 21 and 28 were < 0.05 ppm. Residues in muscle, liver and fat from the 3x and 11x dose groups were $< \text{LOQ}$ (< 0.05 ppm).

OPPTS GLN 860.1400: Magnitude of the Residue in Water, Fish, Irrigated Crops

Diclofop-methyl is not registered for use on aquatic sites; therefore, no residue chemistry data are required under this guideline topic.

OPPTS GLN 860.1460: Magnitude of the Residue in Food-handling Establishments

Diclofop-methyl is not registered for use in food-handling establishments; therefore, no residue chemistry data are required under this guideline topic.

OPPTS GLNs 860.1850/860.1900: Confined/Field Accumulation in Rotational Crops

A confined rotational crop study (MRID 42271901) has been submitted and deemed inadequate because additional sample storage information is required and further efforts to characterize and identify radioactive residues in lettuce and barley forage from the 30-day plant-back interval (PBI) must be conducted. In this study, radioactive residues accumulated at levels >0.01 ppm in the commodities of lettuce, carrots, and barley planted in a sandy loam soil 30 days following application of [¹⁴C]diclofop-methyl at 1x the maximum label rate. The highest radioactive residue levels were found in barley forage (0.101 ppm), and the lowest levels were found in carrot roots and tops (0.025 ppm). At the ~120-day PBI, residues were <0.01 ppm in carrot tops and roots, 0.015 ppm in lettuce, and 0.018-0.056 ppm in barley commodities. At the 365-day PBI, residues were <0.006 ppm in all commodities except barley grain (0.012 ppm).

An adequate degree of characterization was achieved for the 120-day and 365-day crop samples, although no radioactive residues were identified in these samples. At the ~120-day PBI, all extract and hydrolysate fractions from lettuce, carrot, and barley contained <0.01 ppm with the majority of residues (38-83% TRR) being recovered in the residual solids following base hydrolysis. Crop samples from the 365-day PBI were not extracted for analysis as residues in these samples were ≤0.012 ppm.

There are presently no rotational crop restrictions on the diclofop-methyl end-use products registered to AgroEvo USA. The submitted confined rotational crop study is adequate pending submission of sample storage information and storage stability data. The available study supports a 120-day restriction on planting root and tuber vegetables, leafy vegetables and small grains of rotational crops in diclofop-methyl treated soils. If the registrant opts to support a 30-day plant-back interval, identification and characterization of extracted radioactive residues (>10% of the TRR) from lettuce and barley forage are required. Radioactive residues in crop samples should be analyzed as specified in OPPTS GLN 860.1850.

Table A2. Food/Feed Use Patterns Subject To Reregistration for Diclofop-methyl (PC Code 110902, Case 2160).

Site Application Type ^a Application Timing Application Equipment ^a	Formulation [EPA Reg No]	Max. Single Application Rate (lb ai/A)	Max. # Apps./season	Preharvest Interval (Days)	Use Limitations ^b
Barley					
Broadcast application Postemergence at 1-4 leaf stage (spring barley) or following tiller initiation but prior to jointing (winter barley) Ground or aerial equipment	3 lb/gal EC [45639-173] [45639-177 ^c]	1.0	1	66	<p>Use on barley is restricted to the following states: 45639-173: AZ, CO, DE, ID, KS, MD, MN, MT, NE, NV, NM, NC, ND, OK, OR, PA, SC, SD, TX, UT, VA, WA, WV, and WY. 45639-177: AZ, CO, DE, ID, KS, MD, MN, MT, NE, NV, NM, NC, ND, OK, OR, SC, SD, TX, UT, VA, WA, WV, and WY.</p> <p>May be used on all varieties of spring barley and on the following varieties of winter barley: Milton, Boone, Molly Bloom, Wysor, Pennco, Nomini, Anson, Mulligan, Henry, Callio, Starling, and Sussex.</p> <p>Do not apply pre-emergence to barley. Do not apply more than one application per growing season. Do not use a crop oil concentrate in the spray mixture. Do not allow livestock to graze in treated fields and do not harvest forage, hay or straw from fields prior to grain harvest.</p>

Table A2 (continued).

Site Application Type ^a Application Timing Application Equipment ^a	Formulation [EPA Reg No]	Max. Single Application Rate (lb ai/A)	Max. # Apps./season	Preharvest Interval (Days)	Use Limitations ^b
Wheat					
Broadcast application Preplant-incorporated, preemergence, or postemergence prior to development of the first node (jointing) Ground or aerial equipment	3 lb/gal EC [45639-173] [45639-177 ^c]	1.0	1	77	<p>Preplant-incorporated use on winter wheat is restricted to the following states: CO, ID, KS, MT, NE, OR, SD, UT, and WA.</p> <p>Preemergence use on winter wheat is restricted to the following states: 45639-173: AL, AR, DE, GA, IN, KY, LA, MD, MS, MO, NC, OK, OR, PA, SC, TN, TX, VA, WA, and WV. 45639-177: AL, AR, DE, FL, GA, KY, LA, MD, MS, NC, OK, OR, SC, TN, TX, VA, WA, and WV.</p> <p>Postemergence use on winter or spring wheat is allowed in the following states: 45639-173: AL, AR, AZ, CA, CO, DE, GA, ID, IL, IN, KS, KY, LA, MD, MN, MO, MS, MT, NC, ND, NE, NV, NM, OK, OR, PA, SC, SD, TN, TX, UT, VA, WA, and WV. 45639-177: AL, AR, AZ, CO, DE, FL, GA, ID, KS, KY, LA, MD, MN, MS, MT, NC, ND, NE, NV, NM, OK, OR, SC, SD, TN, TX, UT, VA, WA, WV, and WY.</p> <p>Do not apply more than one application per growing season. For postemergence application, up to 1 qt/A of crop oil concentrate may be added to the spray mixture. Do not allow livestock to graze in treated fields and do not harvest forage, hay or straw from fields prior to grain harvest.</p>

^a Minimum application volumes for ground and aerial applications are 10 and 5 gal/A, respectively; applications through irrigation systems are prohibited.

^b The 3 lb/gal EC label specifies a restricted entry interval (REI) of 24 hours.

^c Pending cancellation as per USEPA/OPP Chemical Database (1/13/00).

Table B. Residue Chemistry Science Assessments for Reregistration of Diclofop-methyl (110902).

GLN: Data Requirements	Current Tolerances, ppm [40 CFR]	Must Additional Data Be Submitted?	References ¹	
860.1200: Directions for Use	N/A	Yes ²		
860.1300: Plant Metabolism	N/A	Yes ³	00038848 00064486 00107467 43476902 ³ 43476904 ³ 43995701 ⁴	00038849 00068747 43476901 ³ 43476903 ³ 43476905 ³
860.1300: Animal Metabolism	N/A	No	42450101 ⁵ 43529601 ⁷	43437501 ⁶
860.1340: Residue Analytical Methods				
- Plant commodities	N/A	No	00038972 00149584 42442803 ⁸ 44896101 ⁹	00071381 00155731 42442804 ⁸ 44896102 ⁹
- Animal commodities	N/A	No ¹⁰	44257201 ¹¹ 44178002 ¹²	44178001 ¹²
860.1360: Multiresidue Methods	N/A	No	42272401 ¹³	
860.1380: Storage Stability Data	N/A	No	42442805 ⁸ 44915001 ¹⁴	42857501 ⁸
860.1500: Crop Field Trials				
<u>Legume Vegetables Group</u>				
- Lentils	0.1 [§180.385(a)]	No ¹⁵	00149584	
- Peas, dry	0.1 [§180.385 (a)]	No ¹⁵	00150890	00155731
<u>Cereal Grains Group</u>				
- Barley, grain	0.1 [§180.385(a)]	No	42442802 ⁸	
- Wheat, grain	0.1 [§180.385(a)]	No	42442801 ⁸	

Table B. (continued).

GLN: Data Requirements	Current Tolerances, ppm [40 CFR]	Must Additional Data Be Submitted?	References ¹
<u>Forage, Fodder, and Straw of Cereal Grains Group</u>			
- Barley, hay and straw	0.1	No	42442802 ⁸ 44896102 ⁹
-	[§180.385(a)]		
- Wheat, forage, hay, and straw	0.1	No	42442801 ⁸ 44896101 ⁹
	[§180.385(a)]		
<u>Miscellaneous Commodities</u>			
- Aspirated Grain Fractions (Grain Dust)	None	No	42442801 ⁸
860.1520: Processed Food/Feed			
- Barley	None	No	42442802 ⁸
- Wheat	None	No	42442801 ⁸
860.1480: Meat, Milk, Poultry, and Eggs			
- Fat, Mby, and Meat of Poultry	None	No	44178002 ¹²
- Eggs	None	No	44178002 ¹²
- Milk	None	No	44178001 ¹²
860.1400: Water, Fish, and Irrigated Crops	None	NA	
860.1460: Food Handling	None	NA	
860.1850: Confined Rotational Crops	N/A	Yes ¹⁶	42271901 ¹⁷ 42511601 ¹⁷
860.1900: Field Rotational Crops	None	Reserved ¹⁸	

- References listed were cited in the Diclofop-methyl Phase 4 Review dated 2/12/91, unless otherwise noted.
- The following amendments and clarifications are required for the 3 lb/gal EC formulation (EPA Reg. No. 45639-173):
 - The use of a preemergence application should be clarified. Under the “General Information” section, the product label states “Hoelon® 3EC may be applied preplant-incorporated (to wheat only), preemergence or postemergence”, implying that preemergence application to barley is allowed. However, under the sections for “Preemergence Special Instructions” and the “Special Notes for

Table B. (continued).

- Spring and Winter Barley”, the label prohibits preemergence application of Hoelon 3EC to spring and winter barley.
- The following rotational crop restrictions are required: “Barley and wheat may be rotated at any time. Crops other than barley and wheat may only be rotated at plantback intervals of 120 days.”
3. DP Barcode D210362, S. Funk, 5/19/95.
The HED Metabolism Assessment Review Committee (Memo, 4/7/00, S.Piper) questioned whether the enforcement method would detect metabolites characterized as hydroxy metabolite conjugates (M5 and M7) and indicated that the registrant must demonstrate whether the enforcement method will determine M5 and M7.
 4. DP Barcode D226168, S. Funk, 7/25/96.
 5. DP Barcode D182833, D. Miller, 9/23/93.
 6. DP Barcode D209579, S. Funk, 1/30/96.
 7. DP Barcode D211874, S. Funk, 6/7/95.
 8. DP Barcodes D182054 and D193642, C. Swartz, 8/19/94.
 9. DP Barcode D258888, S. Piper, 10/4/99.
 10. CEB1 will forward Method BL/01/95 version 2 to ACB/BEAD for a method validation.
 11. DP Barcode D235435, S. Piper, 2/24/00.
 12. DP Barcode D232237, S. Piper, 2/29/00.
 13. DP Barcode D177015, L. Edwards, 6/26/92.
 14. DP Barcode D259699, S. Piper, 11/5/99.
 15. Uses on lentils and dry peas have been deleted from the registrant’s label.
 16. Information is required on how long samples were stored at -20 C prior to extraction of ¹⁴C-residues. In addition, solvent extractable ¹⁴C-residues accounting for 22-29% of the TRR (0.013-0.022 ppm) in lettuce and barley forage from the 30-day plant-back interval (PBI) were not sufficiently characterized. However, an adequate degree of characterization was achieved for the 120-day and 365-day crop samples, although no ¹⁴C-residues were identified in these samples. If the registrant provides adequate sample storage information and supporting storage stability data, the available study would support a 120-day restriction on planting of rotational crops in diclofop-methyl treated soils. However, if the registrant opts to support a shorter PBI, then a new confined rotational crop study should be conducted using a representative leafy vegetable, root vegetable and cereal grain planted 30 days following an application of [¹⁴C]diclofop-methyl at 1 lb ai/A. Radioactive residues in crop samples should be analyzed as specified in OPPTS GLN 860.1850.
 17. DP Barcode D200892, S.Piper, 3/21/00.

Table B. (continued).

18. Provided deficiencies in the existing confined rotational crop study are resolved and the registrant amends their label to specify a 120-day PBI for crops other than barley and wheat, then field rotational crop studies will not be required. However, if the registrant intends to support a shorter PBI, then a new confined rotational crop study is required, and the need for field rotational crop studies would be determined after review of the new confined rotational crop study.

TOLERANCE REASSESSMENT SUMMARY

Tolerances for residues of diclofop-methyl are established under 40 CFR §180.385(a). Only tolerances for plant commodities are presently established, and none have been established for animal commodities. Plant commodity tolerances are expressed in terms of the combined residues of the herbicide diclofop-methyl [methyl-2-(4-(2,4-dichlorophenoxy)phenoxy)propanoate] and its metabolites 2-[4-(2,4-dichlorophenoxy)phenoxy]propanoic acid and 2-[4-(2,4-dichloro-5-hydroxyphenoxy)phenoxy]propanoic acid and its conjugates. The chemical structures of diclofop-methyl and its regulated metabolites are depicted in Figure A.

The nature of the residue in plants is based on an acceptable wheat metabolism study. HED reviews of the submitted study concluded that the plant residues of concern requiring regulation should remain the parent, diclofop acid, and hydroxy diclofop and its conjugates provided that food/feed uses of diclofop-methyl are restricted to barley and wheat.

The nature of the residue in animals is based on an acceptable ruminant and poultry metabolism studies. The residues of concern for both ruminants and poultry are diclofop-methyl and diclofop acid, free and conjugated. Regulation of hydroxy diclofop in animal matrices is not necessary since its concentration in animal tissues is relatively low.

We recommend that 40 CFR §180.385(a) should be further subdivided into 40 CFR §180.385(a)(1) and 40 CFR §180.385(a)(2) for separate designations of diclofop residues of concern in plants and animals, respectively.

A summary of diclofop-methyl tolerance reassessment is presented in Table C.

Tolerances To Be Listed Under 40 CFR §180.385(a)(1):

Sufficient field residue data were submitted to reassess the established tolerances for barley grain, barley straw, wheat grain, and wheat straw. There are no registered uses on lentils and peas, and no registrants have committed to support diclofop-methyl uses on these crops; therefore, the established tolerances for these crop commodities should be revoked.

The available barley and wheat processing studies indicate that diclofop-methyl residues of concern do not concentrate in the crop's respective processed fractions. Therefore, tolerances are not required for the processed fractions of barley and wheat.

Tolerances Needed Under 40 CFR §180.385(a)(1):

Tolerances are required and must be proposed for barley hay, wheat forage, and wheat hay. Based on the maximum combined residues from the field trials, we recommend tolerance levels of 6.0 ppm for barley hay, 12.0 ppm for wheat forage, and 1.0 ppm for wheat hay.

Adequate aspirated wheat grain fractions (grain dust) are available; however, based on the use pattern and submitted residue data, a tolerance is not necessary for this RAC.

Tolerances Needed Under 40 CFR §180.385(a)(2):

The available ruminant feeding study suggests that tolerances should be established for the combined residues of diclofop-methyl and diclofop acid (free and conjugated), determined as diclofop methyl, in milk and livestock (cattle, goats, horses, and sheep) commodities. Due to revisions in the Livestock Feed Table (Table II of the Pesticide Assessment Guidelines, Subdivision O), barley hay and wheat forage and hay are now considered animal feed items and residue data are required to establish appropriate tolerances for these commodities. Furthermore, when these commodities are included in the dietary burdens for beef and dairy cattle, consuming feed items treated with diclofop-methyl, the theoretical burdens are 13.8 ppm for beef cattle and 29.5 ppm for dairy cattle. CEB1 notes that the submitted ruminant feeding study (MRID 44178001) did not consider the reassessed tolerances for barley hay and wheat forage when calculating ruminant dietary burdens, as such, the test animals were dosed at levels significantly lower than the 1x, 3x, and 10x levels recommended by Agency's guidelines (860.1480). However, since the submitted study clearly demonstrates transfer of residue to livestock commodities at all fortification levels (0.11 ppm, 0.33 ppm, and 1.1 ppm) and the highest dose (25 ppm) approximates the 1x dietary burden (29.5 ppm), CEB1 recommends that this study be used and that livestock tolerances be determined by extrapolation to the 1x dietary burden. Based on the maximum combined residues observed in milk and tissues of dairy cattle orally administered with the test substance at 25.0 ppm (0.85x maximum dietary burden) and extrapolating to 1x, HED recommend tolerance levels of 4.0 ppm in milk, 7.0 ppm in meat-by-products (excluding kidney), 25.0 ppm in kidney, and 1.0 ppm in meat and fat of cattle, goat, horses, and sheep.

A feeding study on swine is not available. However, translating the residue data from the ruminant feeding study and using the 0.09 ppm maximum theoretical dietary burden for swine, we conclude that tolerances should be established for diclofop-methyl residues in fat and meat-by-products (mbyp) of hogs. Tolerances at the LOQ (0.05 ppm) should be established for residues in hog fat and mbyp (excluding kidney), and a separate tolerance should be established at 0.1 ppm for residues in hog kidney. Tolerances are not required for the meat of hogs as residues were <LOQ in meat of cattle dosed at a level (1.1 ppm) equivalent to 12x the maximum dietary burden for hogs.

Table C. Tolerance Reassessment Summary for Diclofop-Methyl.

Commodity	Current Tolerance (ppm)	Tolerance Reassessment (ppm)	Comment/Correct Commodity Definition
Tolerances To Be Listed under 40 CFR §180.385(a)(1):			
Barley, grain	0.1	0.1	
Barley, straw	0.1	0.1	
Lentils	0.1	Revoke	Uses on lentils and dry peas have been deleted from the registrant's label.
Pea seeds, dry	0.1	Revoke	
Wheat, grain	0.1	0.1	
Wheat, straw	0.1	0.1	
Tolerances Needed Under 40 CFR §180.385(a)(1):			
Barley, hay	None	6.0	
Wheat, forage	None	12.0	
Wheat, hay	None	1.0	
Tolerances Needed Under 40 CFR §180.385(a)(2):			
Cattle, fat	None	1.0	
Cattle, meat		1.0	
Cattle, mbyp (excluding kidney)		7.0	
Cattle, kidney		25.0	
Goat, fat	None	1.0	
Goat, meat		1.0	
Goat, mbyp (excluding kidney)		7.0	
Goat, kidney		25.0	
Hog, fat	None	0.05	
Hog, mbyp (excluding kidney)		0.05	
Hog, kidney		0.1	
Horse, fat	None	1.0	
Horse, meat		1.0	
Horse, mbyp (excluding kidney)		7.0	
Horse, kidney		25.0	
Milk	None	4.0	
Sheep, fat	None	1.0	
Sheep, meat		1.0	
Sheep, mbyp (excluding kidney)		7.0	
Sheep, kidney		25.0	

No maximum residue limits (MRLs) for diclofop-methyl have been established or proposed by Codex for any agricultural commodity. Therefore, no compatibility questions exist with respect to U.S. tolerances.

AGENCY MEMORANDA CITED IN THIS DOCUMENT

DP Barcode: D177015
Subject: Diclofop-Methyl, Diclofop Free Acid, and the Phenoxyhydroxy Metabolite of
Diclofop-methyl. Case # 2160. Multi-residue Methods Protocol.
From: L. Edwards
To: H. Hundley
Dated: 6/26/92
MRID(s): 42272401

DP Barcode: D182833
Subject: Diclofop-Methyl. Ruminant Metabolism Study in Goats (milk, fat, muscle, liver,
kidney, and blood).
From: D. Miller
To: T. Luminello
Dated: 9/23/93
MRID(s): 42450101

DP Barcode: D182054 and D193642
Subject: Diclofop-Methyl. List B Reregistration Case 2160/Chemical ID No. 110902.
Review of Barley and Wheat Field Trials, Processing Studies, and Storage Stability
Data [Guideline Ref. Nos. 171-4(k), 171-4(l), and 171-4(e)].
From: C. Swartz
To: K. Davis
Dated: 8/19/94
MRID(s): 42442801 through 42442805 and 42857501

DP Barcode: D210362
Subject: Diclofop-Methyl. (Case 2160, Chemical 110902, List B). Nature of the Residue
in Wheat (GLN 171-4(a)).
From: S. Funk
To: K. Davis\T. Luminello
Dated: 5/19/95
MRID(s): 43476901 through 43476905

DP Barcode: D211874
Subject: Diclofop-Methyl (Case 2160, Chemical 110902, List B). Nature of the Residue in Poultry (GLN 171-4(b)).
From: S. Funk
To: K. Davis\T. Luminello
Dated: 6/7/95
MRID(s): 43529601

DP Barcode: D209579
Subject: Diclofop-Methyl (Case 2160, Chemical 110902, List B). Nature of the Residue in Ruminants (GLN 171-4(b)). AgrEvo (Hoechst and NOR-AM) Study Upgrade.
From: S. Funk
To: K. Davis\T. Luminello
Dated: 1/30/96
MRID(s): 43437501

DP Barcode: D226168
Subject: Diclofop-Methyl (Case 2160, Chemical 110902, List B). Nature of the Residue in Wheat (GLN 171-4(a)). Additional Data from AgrEvo USA (Hoechst/Schering).
From: S. Funk
To: P. Deschamp
Dated: 7/25/96
MRID(s): 43995701

DP Barcode: D258888
Subject: Diclofop-Methyl Reregistration Case No. 2160. PC Code 110902. Magnitude of the Residue of Diclofop-Methyl in Wheat and Barley.
From: S. Piper
To: T. Peterson/C. Jarvis
Dated: 10/4/99
MRID(s): 44896101 and 44896102

DP Barcode: D259699
Subject: Diclofop-Methyl Reregistration Case No. 2160. PC Code 110902. Stability of Diclofop-methyl and Diclofop-acid in Beef Muscle, Whole Milk, Chicken Livers, and Eggs During Frozen Storage.
From: S. Piper
To: T. Peterson/C. Jarvis
Dated: 11/5/99
MRID(s): 44915001

DP Barcode: D235435
Subject: Diclofop-Methyl. Independent Laboratory Validation of Proposed GC/ECD Enforcement Method for Animal Commodities.
From: S. Piper
To: T. Peterson/C. Jarvis
Dated: 2/24/00
MRID(s): 44257201

DP Barcode: D232237
Subject: Diclofop-Methyl. Ruminants and Poultry Feeding Study for Diclofop-Methyl.
From: S. Piper
To: T. Peterson/C. Jarvis
Dated: 2/29/00
MRID(s): 44178001 and 44178002

DP Barcode: D200892
Subject: Diclofop-Methyl. Accumulation of Residues in Rotational Crops.
From: S. Piper
To: T. Peterson/C. Jarvis
Dated: 3/21/00
MRID(s): 42271901 and 42511601

RESIDUE CHEMISTRY CITATIONS

00038848 Thier, W.; Schulze, E.F.; Kocher, H.; et al. (1975) Information on the Decomposition of HOE 23408 OH in Wheat under Open Field Conditions: Report 238/75. (Translation; unpublished study received Aug 22, 1975 under 6G1664; prepared by Hoechst, AG, submitted by American Hoechst Corp., Somerville, N.J.; CDL:094680-F)

00038849 Gorbach, S.; Asshauer, J.; Kunzler, K. (1975) Concerning the Identity of Breakdown Products of the Herbicide HOE-23408 OH in Wheat: Report No. 239/75. (Translation; unpublished study received Aug 22, 1975 under 6G1664; prepared by Hoechst, AG, submitted by American Hoechst Corp., Somerville, N.J.; CDL:094680-G)

00038972 Beam, L. (1977) Analytical Method: Determination of Hoe-23408 and Its Hydroxy Metabolite (Hoe-35449) in the Grain, Straw and Forage of Wheat and Barley: Laboratory No. 6E-4200; A10104. Method dated Jan 14, 1977. (Unpublished study received Aug 11, 1977 under 8340-EX-1; prepared by Cannon Laboratories, Inc., submitted by American Hoechst Corp., Somerville, N.J.; CDL:096311-Q)

00064486 Gorbach, S.; Asshauer, J.; Kunzler, K. (1975) Concerning the Identity of Breakdown Products of the Herbicide HOE-23408 OH in Wheat: Report 239/75; Report A03606. (Unpublished study received Nov 4, 1980 under NC 80/25; prepared by Hoechst, AG, submitted by North Carolina, Dept. of Agriculture, Pesticide and Plant Protection Div. for American Hoechst Corp., Somerville, N.J.; CDL:243742-D)

00068747 Gorbach, S.; Kunzler, K.; Muller, H.J. (1976) Report on Residues of HOE 23408 OH and Its Degradation Products in Soybeans: Bericht-Nr. (B) 23/76. (Unpublished study received Jul 22, 1976 under 8340-EX-1; prepared by Hoechst AG, West Germany, submitted by American Hoechst Corp., Somerville, N.J.; CDL:095503-I)

00071381 Hormby, T. (1979) Analysis of HOE 23408, HOE 35449 and the Phenol Metabolite of HOE 23408 in Crops and Soil: Laboratory No. 9PL-72-MT. Method dated Jun 20, 1979. (Unpublished study received Mar 2, 1981 under 8340-11; prepared by Biospherics, Inc., submitted by American Hoechst Corp., Somerville, N.J.; CDL:244464-C)

00107467 Shimabukuro, R.; Walsh, W.; Hoerauf, A. (1978) Metabolism and Selectivity of Diclofop-methyl in Wild Oat and Wheat. (U.S. Dept. of Agriculture, Science and Education Administration, Agricultural Research, Metabolism and Radiation Research Laboratory; unpublished study; CDL:098356-D)

00149584 American Hoechst Corp. (1985) Residue Chemistry: Hoe-23408: Hoelon Herbicide on Flax Seed . Unpublished compilation. 698 p.

00150890 American Hoechst Corp. (1984) Residue Chemistry Data from Dry Peas and Lentils

after Hoelon Use. Unpublished compilation. 369 p.

00155731 American Hoechst Corporation (1985) Hoelon 3EC Herbicide Application for Full Registration and Tolerances on Dry Peas: Section D-Residue Chemistry. Unpublished compilation. 301 p.

42271901 Erstfeld, K.; Eschbach, J.; Lange, B. (1992) Diclofop-methyl [carbon 14]: Confined Accumulation in Rotational Crops 30- and 120-Day Experiment: 120-Day Experiment: Lab Project Number: 89-0159: 90-0099: EF-89-31. Unpublished study prepared by Pan-Ag. Labs and Bio/dynamics Inc. 213 p.

42272401 Ver Hey, M. (1992) Food and Drug Administration Pesticide Analytical Manual Volume 1. Testing of Diclofop-methyl (HOE-023408), HOE-021079 (Diclofop Free Acid) and HOE-038182 (the Phenoxyhydroxy Metabolite of Diclofop-methyl): Lab Project Number: HOECHST-1148. Unpublished study prepared by Colorado Analytical Research & Development Corp. 456 p.

42442801 PDMS citation not available to Dynamac reviewer.

42442802 PDMS citation not available to Dynamac reviewer.

42442803 Niedzwiadek, B. (1992) Diclofop-Methyl Residue Method Validation in Wheat Grain, Wheat Straw, Barley Grain, and Barley Straw Matrices: Lab Project Number: XM-11: HOELON. Unpublished study prepared by Xenos Laboratories, Inc. 106 p.

42442804 Niedzwiadek, B. (1992) Diclofop-Methyl Residue Method Validation in Wheat Grain and Barley Grain Process Fractions: Lab Project Number: XEN91-24B. Unpublished study prepared by Xenos Laboratories, Inc. 107 p.

42442805 Niedzwiadek, B.; Czarnecki, J. (1992) Diclofop-Methyl Storage Stability in Wheat: Lab Project Number: XEN91-24. Unpublished study prepared by Xenos Laboratories, Inc. 111 p.

42450101 Struble, C. (1992) Nature of Residue of [carbon 14]-Diclofop-methyl in Lactating Goats: Lab Project Number: HWI 6187-124. Unpublished study prepared HOELON. Number: HWI 6187-124. Unpublished study prepared by Hazleton Wisconsin, Inc. 188 p.

42511601 Erstfeld, K.; Fischer, R.; O'Grodnick, J. et al. (1992) (Carbon 14)-Diclofop-methyl: Confined Accumulation in Rotational Crops: Lab Project Number: 89-0159: EF-89-31: 90-0099. Unpublished study prepared by Pan-Agricultural Labs, Inc. and Bio/dynamics Inc. 110 p.

42857501 Bertrand, N.; Czarnecki, J. (1993) Diclofop-methyl: Storage Stability in Wheat: Supplemental Study: Lab Project Number: XEN 91-24. Unpublished study prepared by Xenos

Labs, Inc. 43 p.

43437501 Struble, C. (1994) Registrant's Response to the Agency's Review of the Diclofop-methyl Ruminant Metabolism in Lactating Goats: Lab Project Number: 6187-124: HWI 6187-124. Unpublished study prepared by Hazleton Wisconsin, Inc. 50 p.

43476901 PDMS citation not available to Dynamac reviewer.

43476902 Gildemeister, H. (1994) HOE 023408 (Dichlorophenyl-(carbon 14)) Metabolism in Wheat (*Triticum aestivum*): Lab Project Number: CM 92/059: A 52693: A52693. Unpublished study prepared by Hoechst Schering AgrEvo GmbH. 40 p.

43476903 Gildemeister, H. (1994) HOE 023408 (Dichlorophenyl-(carbon 14)) Metabolism in Wheat (*Triticum aestivum*): Addendum to Report CM92/059: Lab Project Numbers: CM92/059: A 53314: CM 92/059. Unpublished study prepared by Hoechst Schering AgrEvo GmbH. 11 p.

43476904 Gildemeister, H. (1994) HOE 023408 (Dichlorophenyl-(carbon 14)) Further Investigations of the Metabolism in Wheat (*Triticum aestivum*): Using Samples of Study CM92/059: Lab Project Number: CM94/076: CM 94/076: A 53315. Unpublished study prepared by Hoechst Schering AgrEvo GmbH. 21 p.

43476905 Gildemeister, H. (1994) HOE 023408 (Dichlorophenyl-(carbon 14)): Metabolism in Wheat (*Triticum aestivum*): Lab Project Numbers: CM92/0690: CM 92/0690: A 53316. Unpublished study prepared by Hoechst Schering AgrEvo GmbH. 41 p.

43529601 Struble, C. (1995) Nature of the Residue of (carbon 14)-Diclofop-methyl in Laying Hens: Lab Project Number: HWI 6187-131. Unpublished study prepared by Hazleton Wisconsin, Inc. 242 p.

43995701 Buerkle, W. (1996) Diclofop-methyl, Hoe 023408 Dioxyphenyl-u-(carbon 14)-Labeled Code: Hoe 023408 00 ZE980007: Wheat Metabolism, Additional Investigations Concerning Existing Study, MRID #43476901-05: Lab Project Number: CM95/104: A 56512. Unpublished study prepared by Hoechst Schering AgrEvo GmbH. 51 p.

44178001 Dacus, S. (1996) Diclofop-methyl Derived Residues in the Meat and Milk of Dairy Cows Resulting from Oral Ingestion of Diclofop-methyl: Lab Project Number: A 55826: BL-95R-02: 149-005-10. Unpublished study prepared by AgrEvo USA Co. 137 p. Relates to letter L0000165.

44178002 Dacus, S. (1996) Diclofop-methyl Derived Residues in the Eggs and Tissues of Laying Hens Resulting from Oral Ingestion of Diclofop-methyl: Lab Project Number: A 55825: BL-95R-03: 149-004-09. Unpublished study prepared by AgrEvo USA Co. 116 p.

44257201 Barker, W. (1997) Independent Method Validation Ruggedness Trial for the Determination of Diclofop-Methyl (HOE-023408) and Its Metabolite Diclofop Acid (HOE-021079) in Poultry Tissues, Eggs, Beef Tissues, and Milk Using AgrEvo Method No. BL/01/95, Entitled "Determination of Diclofop-Methyl (HOE-023408) and Diclofop Free Acid (HOE-021079) in Beef and Chicken Tissues, Meat By-Products, Milk, and Eggs": Final Report: Lab Project 00107463 American Hoechst Corp. (1979) HOE-23408--Soybeans. (Compilation; unpublished study received Jun 27, 1979 under 8F2110; CDL: 098355-A)

44896101 Singer, S. (1999) At-Harvest Diclofop-methyl Derived Residues in Wheat (Forage and Hay) Following a Single Application of HOELON 3EC at the Maximum Permitted Rate USA, 1997 and 1998: Lab Project Number: BLR96R001: B002199: AE F023408OHEC36B800. Unpublished study prepared by AgrEvo USA Company. 169 p.

44896102 Singer, S. (1999) At-Harvest Diclofop-methyl Derived Residues in Barley Hay Following a Single Application of HOELON 3EC at Maximum Permitted Rate USA, 1997 and 1998: Lab Project Number: BLR96R002: AEF023408OHEC36B800: B002200. Unpublished study prepared by AgrEvo USA Company. 119 p.

44915001 Dacus, S. (1999) Stability of Diclofop Methyl(AE F023408) and Diclofop Acid (AE F021709) in Beef Muscle, Whole Milk, Chicken Livers, and Eggs During Frozen Storage, USA, 1996: Lab Project Number: BL96R04: C004149. Unpublished study prepared by AgrEvo USA Company. 65 p.